Docket No. 192376US0



**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

IN RE APPLICATION OF: Uwe ERNST, et al.

SERIAL NO: 09/580,526 GAU: 1724

FILED: May 30, 2000 EXAMINER:

FOR: PROCESS FOR ADSORPTION OF ORGANIC COMPOUNDS, AND ADSORBENTS OF PULVERULENT RUBBER



# INFORMATION DISCLOSURE/RELATED CASE STATEMENT UNDER 37 CFR 1.97

ASSISTANT COMMISSIONER FOR PATENTS
WASHINGTON, D.C. 20231

SIR:

Applicant(s) wish to disclose the following information.

## REFERENCES

- ☐ The applicant(s) wish to make of record the references listed on the attached form PTO-1449. Copies of the listed references are attached, where required, as are either statements of relevancy or any readily available English translations of pertinent portions of any non-English language references.
- ☐ A check is attached in the amount required under 37 CFR §1.17(p).

## RELATED CASES

- ■ Attached is a list of applicant's pending application(s) or issued patent(s) which may be related to the present application. A copy of the claims and drawings of the pending application(s) is attached.
- ☐ A check is attached in the amount required under 37 CFR §1.17(p).

## CERTIFICATION

- ☐ Each item of information contained in this information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of this statement.
- ☐ No item of information contained in this information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application or, to the knowledge of the undersigned, having made reasonable inquiry, was known to any individual designated in 37 CFR §1.56(c) more than three months prior to the filing of this statement.

## DEPOSIT ACCOUNT

- ■ Please charge any additional fees for the papers being filed herewith and for which no check is enclosed herewith, or credit any overpayment to deposit account number 15-0030. A duplicate copy of this sheet is enclosed.

Respectfully submitted,

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## LIST OF RELATED CASES

| Docket Number | Serial or Patent No. | Filing or Issue Date | Status or Patentee |
|---|---|---|---|
| 199917US0 | 09/791,622 | 02/26/01 | PENDING |
| 192376US0* | 09/580,526 | 05/30/00 | PENDING |

*Present application; listed for information.

WHAT IS CLAIMED IS:

1. A process for preparing a fine-particle, pulverulent, filled rubber, comprising:

precipitating said fine-particle, pulverulent, filled rubber from an aqueous mixture of a filler suspension and a rubber-latex emulsion in the absence of a water-soluble salt of a metal of Groups IIa, IIb, IIIa or VIII of the Periodic Table of the Elements, and in the absence of an alkali metal silicate.

2. The process according to Claim 1, further comprising:

dividing an entirety of a filler into core black, splitting black 1 and splitting black 2 wherein an amount of water-insoluble constituent of the filler is from 1 to 10%;

dispersing the core black in water, thereby providing a suspension of the core black;

dispersing the splitting black 1 in water, thereby providing a suspension of the splitting black 1;

dispersing the splitting black 2 in water, thereby providing a suspension of the splitting black 2;

adjusting the pH to a value of from 2.5 to 6 by using a Brönsted acid;

running said suspension of the core black together with said rubber latex emulsion through a tubular reactor, thereby initiating the coagulation of rubber powder grains;

forming said rubber powder grains;

collecting said rubber powder grains in a tank and simultaneously continuously feeding said suspension of said splitting black 1 having a pH of from 7 to 9.5 to the rubber powder grains collected in said tank, thereby controlling the grain size of the rubber powder;

continuously maintaining the pH of the rubber powder suspension;

adding said suspension of splitting black 2 to the aqueous rubber powder suspension

after the precipitation, wherein optionally an acid is used to maintain the pH at a value of from 2.5 to 6.

3. The process according to Claim 2, wherein said entirety of said filler comprises of from 30 to 60% by weight of core black, of from 30 to 60% by weight of splitting black 1 and of from 5 to 15% by weight of splitting black 2.

4. The process according to Claim 2, wherein said Brönsted acid is sulfuric acid.

5. The process according to Claim 1, further comprising:

dispersing 50 to 98% by weight of an entirety of a filler in water, thereby providing said filler suspension;

wherein a solids content in said aqueous suspension is from 1 to 10% and wherein the pH is from 7 to 9.5;

forming an initial charge of said filler suspension with said rubber-latex emulsion in a precipitation tank under vigorous stirring;

precipitating said fine-particle, pulverulent, filled rubber by adding an acid;

adjusting the pH of the suspension to a value of from 2.5 to 6;

suspending the remaining 2 - 50% by weight of said entirety of said filler with a solids content of from 1 to 10% in water;

adding the suspension of the remaining 2 - 50% by weight of said entirety of said filler, wherein optionally the pH is adjusted.

6. The process according to Claim 5, wherein said acid sulfuric acid.

7. The process according to Claim 1, wherein

said precipitating occurs at temperatures of from 10 to 60°C.

8. The process according to Claim 1, wherein said rubber is selected from the group consisting of emulsion SBR, acrylonitrile rubber, aqueous-emulsion-polymerized butadiene

rubber, natural rubber and a mixture thereof.

9. The process according to Claim 8, wherein said natural rubber comprises field latex with a Mooney viscosity of from 50 to 120 Mooney units.

10. The process according to Claim 1, wherein a solids content of said rubber-latex emulsion is from 15 to 65%.

11. The process according to Claim 2 or 5, wherein the filler comprises a carbon black.

12. The process according to Claim 11, wherein the carbon black is selected from the group consisting of furnace black, gas black, flame black and mixtures thereof;

wherein said carbon black has an iodine adsorption value of from 5 to 1000 $m^2/g$, a CTAB number of from 15 to 600 $m^2/g$, a DBP adsorption of from 30 to 400 ml/100 g and a 24 M4 DBP number of from 50 to 370 ml/100 g; and

wherein an amount of said carbon black is from 5 to 250 parts by weight based on 100 parts by weight of rubber.

13. The process according to Claim 11, wherein said carbon black has improved dynamic properties and is used in an amount of from 20 to 250 parts by weight based on 100 parts by weight of rubber.

14. The process according to Claim 1, wherein said fine-particle, pulverulent, filled rubber comprises processing or vulcanizing auxiliaries selected from the group consisting of zinc oxide, zinc stearate, stearic acid, a polyalcohol, a polyamine, a plasticizer, an aging inhibitor, a reinforcing resin, a flame retardant, a pigment, a crosslinking agent, sulfur and mixture thereof.

15. The process according to Claims 2 or 5, further comprising dewatering said rubber powder by a centrifuge or a vacuum-belt filter.

16. The process according to Claim 15, further comprising drying of said rubber powder using heat to a residual moisture of $\leq 3\%$.

17. The process according to Claim 16, further comprising dry-coating of said rubber powder.

18. The process according to Claim 17, wherein from 0.1 to 3 phr of a coating material is applied to said rubber powder in a powder mixer.

19. The process according to Claim 17, wherein said coating material is selected from the group consisting of stearic acid, silica, zinc oxide or a mixture thereof.

20. A fine-particle, pulverulent, filled rubber obtained by the process of Claim 1.

21. A vulcanizable rubber mixture, comprising the fine-particle, pulverulent, filled rubber as claimed in Claim 20.

## ABSTRACT

Fine-particle, filled rubber powders prepared by precipitation from aqueous mixtures of a filler suspension and a rubber-latex emulsion in the absence of water-soluble salts of metals of groups IIa, IIb, IIIa, or VIII of the Periodic Table of the Elements and in the absence of alkali metal silicate are used for vulcanizable rubber mixtures.


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